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(21) International Application Number: PCT/US97/19812 (22) International Filing Date: 30 October 1997 (30.10.97) (30) Priority Data: 60/029,460 30 October 1996 (30.10.96) US (71) Applicant (for all designated States except US): TARREN WOOD PRODUCTS, INC. [US/US]; 15551 Greenock Lane, Fort Myers, FL 33912 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): GRANTHAM, Robert [US/US]; 15551 Greenock Lane, Fort Myers, FL 33912 (US). WARREN, John, S., Jr. [US/US]; 4000 Oak Branch Court, Mobile, AL 36693 (US). (74) Agents: ORKIN, Russell, D. et al.; Webb Ziesenheim Bruening Logsdon Orkin & Hanson, P.C., 700 Koppers Building, 436 Seventh Avenue, Pittsburgh, PA 15219-1818 (US).		(81) Designated States: AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: METHOD OF PRESSURE TREATING BOARDS		
(57) Abstract		
<p>A method for pressure treating wood products is provided wherein wood products are pressure treated in an aqueous solution of a silicate, a rheology modifier, a wetting agent and, optionally, borax; the aqueous solution having a pH of at least 9.0. A suitable dye can be added to the aqueous solution to color the wood product. The present invention is also directed to an aqueous solution for pressure treating wood and a wood product prepared according to the method of the present invention.</p>		

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METHOD OF PRESSURE TREATING BOARDS

BACKGROUND OF THE INVENTION1. Field of the Invention

5 The present invention is directed to a formaldehyde-free method for treating wood products, especially green lumber, to strengthen the wood products and to render the wood products flame retardant and resistant to moisture and insects.

2. Description of the Related Art

10 Prior art publications disclose treatment of wood products with chemicals such as silicates, creosote, copper naphthenate, pentachlorophenol and chromated copper arsenate to preserve the wood products and to render them fire-retardant and insect repellent. After treatment, the
15 wood products can be painted, soaked or pressure treated to coat and/or impregnate the wood products.

United States Patent No. 3,974,318 to Lilla, discloses such a process. In the Lilla patent, a water soluble silicate composition is applied to a wood product
20 and the product is subsequently treated with a water soluble metallic salt compound to form a water insoluble metallic silicate in the wood product.

Although the prior art methods and compositions are suitable for treating dried lumber and manufactured
25 wood products, no products or methods have been found suitable for treating green lumber or other wood products having a high water content.

It is, therefore, an object of the present invention to provide a wood product treatment method and
30 compound suitable therefor for treating green lumber as well as to provide a superior method and composition for completely treating dried lumber and manufactured wood products.

SUMMARY OF THE INVENTION

35 The present invention is directed to a method and a composition for treating wood products as well as a treated wood product. The method provides for treating the wood products with a silicate based solution that will

fully penetrate and preserve wood products, including green lumber. The method includes pressure treating the wood products in an aqueous solution of sodium silicate which further includes a wetting agent, a rheology modifier and, preferably, borax, at a pH of at least 9.0. Addition of the rheology modifier and the wetting agent to the solution allows the chemicals to fully penetrate the wood product irrespective of whether the wood product is green lumber or a dried and/or processed wood product.

The composition of the present invention is an aqueous solution having a pH of at least 9.0 and including silicate, a wetting agent and a rheology modifier, the solution preferably includes borax. The method of the present invention includes the following steps: placing the material in a pressure vessel and applying a vacuum; contacting the material with an aqueous solution and increasing the pressure in the pressure vessel; draining the aqueous solution and reducing the pressure, preferably, applying a vacuum; and drying the treated wood product. The present invention is also directed to pressure treated wood product prepared by the above-described method.

BRIEF DESCRIPTION OF THE DRAWINGS

Figs. 1a-c are graphic representations of flame spread distance, temperature and smoke developed versus time in testing of 5/8" oriented strand board (OSB) prepared according to the method of the present invention.

Figs. 2a-c are graphic representations of flame spread distance, temperature and smoke developed versus time in testing of 3/4" particle board prepared according to the method of the present invention.

Figs. 3a-c are graphic representations of flame spread distance, temperature and smoke developed versus time in testing of 5/8" douglas fir plywood prepared according to the method of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention is directed to a method and composition for treatment of wood products which preserves

the wood products and renders the wood products fire, moisture and insect resistant. The term "wood products" refers generally to products derived from wood, but is not exclusively limited to oriented strand board, medium-density fiber board (MDF), plywood, particle board, paper products and natural wood products, including both green and dried lumber. The treated wood products are generically useful for construction purposes. More specifically, for purpose of example only, the treated products may be used in flooring, fire doors, exterior beams and columns, fire panel materials and sheeting, exterior sheeting including siding, cabinet manufacturing, furniture manufacturing, railroad cross ties, landscape timbers, floor plating, fire-retardant lumber, door jams, sea walls, countertops, exterior fascia material, and in window manufacturing.

The method of the present invention utilizes an aqueous solution for treating the wood products which includes a silicate, a wetting agent and a rheology modifier. The pH of the solution is preferably greater than 9.0. The solution most preferably includes borax as an insecticidal agent. Solutions of this type are commercially available under the trade designations WOF-24 TWF-27 and TWF-27B.

As described above, the aqueous solution is a solution of sodium silicate, a wetting agent, and a rheology modifier and preferably includes borax. The relative quantities of components of the aqueous solution may be varied, the ranges of the components of the solution are preferably between 18% and 60% sodium silicate, 0.01% and 2% wetting agent and 0.01% to 2% rheology modifier and, optionally, 1% and 10% borax. The remainder of the solution includes water and any other desirable additive such as a suitable dye or staining agent. The aqueous solution is most preferably 61.8% water, 38% sodium silicate, 0.1% wetting agent and 0.1% rheology modifier and, optionally, including 5% borax (with 56.8% water).

Examples of rheology modifiers include, but are not limited to, thickening agents including cellulosic agents, such as hydroxymethyl cellulose and carboxymethyl cellulose, and acrylic thickeners. Examples of suitable wetting agent include but are not limited to metasodium silicate, sodium dodecyl sulfate and sodium lauryl sulfate. The aqueous solution is preferably prepared having 21% solids, or active ingredients as measured by refractive index. In use, the aqueous solution may be diluted to range between 0 and 21% solids or active ingredients. The suitable dilution for a given wood product is determined empirically. For instance, it was empirically determined that MDF and particle board are best treated with a solution of 8% solids while railroad ties (red and white oak) are best treated with a solution of 12% solids.

To treat the wood products according to the present invention, the wood product is placed in a pressure vessel. The door of the pressure vessel is closed, and a vacuum of approximately 35 pounds is applied. The chamber is flooded with the aqueous solution described above.

A pressure of 40 to 160 pounds is applied to the wood products in the aqueous solution. Most preferably, pressure of 40 pounds is applied for particle board, MDF and OSB and a pressure of 140 pounds is applied for natural wood products. During pressure treatment, the aqueous solution is circulated under pressure for between 20 and 90 minutes. The wood products are preferably treated for 30 minutes in the case of particle board, MDF and OSB and 90 minutes in the case of natural wood products. The chamber is drained and a vacuum of about 30 pounds is held for 10 minutes. The chamber is opened and the treated wood product is removed. The treated wood product is then either placed in a drying kiln and slow-dried for 24 hours with hot air and steam, air dried for 30 days or microwave dried for up to 8 hours.

It is believed, without any intention of being bound to the theory, that the treatment causes a molecular

bonding of the silicate to the cellulose molecules in the wood and changes the wood product's pH. The molecular change renders the wood product fire-retardant and resistant to rotting, the pH change and addition of borax renders the wood product resistant to insect infestation. The treatment process is complete throughout the board.

EXAMPLES

The fire-retardant ability of this process was evaluated according to the following procedure by the Hardwood Plywood and Veneer Association Laboratory and Testing Service, a commercial testing service, in accordance with ASTM designation E 84, "Standard Test Method for Surface Burning Characteristics of Building Materials." This 25-foot tunnel method is also described by NFPA No. 255 and UL No. 723.

The purpose of the test is to determine the relative performance of the test material under standardized fire exposure. Results are given for flame spread and smoke developed. The values obtained from burning the test material represent a comparison with that of 1/4" inorganic reinforced cement board expressed as zero and red oak flooring expressed as 100. The furnace was preheated to a minimum of 150°F as measured by an 18 AWG thermocouple embedded in cement 1/8" below the floor surface of the chamber, 23-1/4 feet from the center line of the ignition burners. The furnace is then cooled to 105°F ($\pm 5^\circ\text{F}$) as measured by a thermocouple embedded 1/8" below the floor surface of the test chamber 13 feet from the fire end.

Prior 10-minute tests with 1/4" inorganic reinforced cement board provided the zero reference for flame spread. Periodic 10-minute tests with unfinished select grade red oak flooring provided for the 100 reference for flame spread and smoke development.

The wood products tested in the following examples were treated in the following manner. The material was placed in a pressure vessel. The door was

then closed, and a vacuum of 35 pounds was achieved. The chamber was flooded with a 32% volume solution of WOF-24, TWF-27 or TWF-27B to a pressure of 110 pounds. The solution was circulated for 30 minutes. The chamber was
5 drained, and vacuum of 30 pounds was pulled and held for 10 minutes. The chamber was then returned to ambient atmospheric pressure. The chamber was opened and the treated wood product was removed and placed in a drying kiln where it was slow-dried for 24 hours with hot air and
10 steam.

Example 1: WOF-24 Pressure-Treated 5/8" OSB

A 5/8" OSB test sample was prepared and tested according to the procedures described above. The test results are as follows:

15 Material Tested

- | | | |
|----|-----|--|
| | 1) | Manufacturer: World Environmental
Irvington, Alabama |
| | 2) | Burn Number 1 |
| | 3) | Average Thickness (in.) .547 |
| 20 | 4) | Average Weight (lbs./sq.ft.) 2.38 |
| | 5) | Average Groove Depth (in.) N/A |
| | 6) | Product Description: WOF-24 pressure-treated
5/8" oriented strand board
Treatment concentration: 33%
WOF-24 |
| 25 | 7) | Color Brown |
| | 8) | Surface Both sides treated |
| | 9) | Sample Selection Manufacturer |
| | 10) | Date of Selection 4/1996 |
| 30 | 11) | Material Description By Manufacturer |
| | 12) | Method of Mounting Self-supporting on
ledges |
| | 13) | Sample Conditioning (days) 22 |

Test Conditions and Data

- | | | |
|----|----|-----------------------------------|
| 35 | 1) | Specimen Preheat Time (min.) 2:00 |
| | 2) | Tunnel Brick Temp. (deg. F) 105 |
| | 3) | Ignition Time (seconds) 55 |

- 4) Time to End of Tunnel
or Flamefront Distance 13' @ 10:00
- 5) Time-Distance Curve Area
(min./ft.) 46.9
- 5 6) Fuel and Temperature
- a) Fuel (cu.ft./min.) 4.946
- b) Max. Vent End Temp.
(deg. F) 680
- 10 c) Time to Max. Temp.
(min.) 9:55
- 7) After Flaming No

Test Results

Test results calculated on the basis of the areas under the curves of flame spread distance, temperature, and smoke developed versus time, as shown in Figs. 1a-c, are provided in the table below for calibration materials and for:

WOF-24 Pressure Treated 5/8" Oriented Strand Board

<u>Material Description</u>	<u>Flame Spread</u>	<u>Fuel Contributed</u>	<u>Smoke Developed Index</u>
High-density inorganic reinforced cement board	0	0	0
Red oak flooring	100	100	100
Test Sample	25	20	15

OBSERVATIONS: No burnthrough. Surface charred and flaky to 11-1/2 feet.

REMARKS: The average moisture content of the first panel was 15.2%.

CONCLUSIONS: Based on one test, the flame spread, calculated according to ASTM E-84-94, meets Class A (Class I) - 25 or under flame spread.

Example 2: WOF-24 Pressure-Treated 3/4" Particle Board

A 3/4" particle board test sample was prepared and tested according to the procedures described above. The test results are as follows:

5	<u>Material Tested</u>	
	1) Manufacturer:	World Environmental Irvington, Alabama
	2) Burn Number	1
	3) Average Thickness (in.)	.824
10	4) Average Weight (lbs./sq.ft.)	3.76
	5) Average Groove Depth (in.)	N/A
	6) Product Description:	WOF-24 TWP-27 and TWP-27B pressure-treated 3/4" particle board
15		Treatment concentration: 12% WOF-24, TWP-27 and TWP-27B
	7) Color	Brown
	8) Surface	Both sides treated
	9) Sample Selection	Manufacturer
20	10) Date of Selection	4/1996
	11) Material Description By	Manufacturer
	12) Method of Mounting	Self-supporting on ledges
	13) Sample Conditioning (days)	22
25	<u>Test Conditions and Data</u>	
	1) Specimen Preheat Time (min.)	2:00
	2) Tunnel Brick Temp. (deg. F)	105
	3) Ignition Time (seconds)	90
	4) Time to End of Tunnel	
30	or Flamefront Distance	11' @ 7:30
	5) Time-Distance Curve Area (min./ft.)	39.2
	6) Fuel and Temperature	
	a) Fuel (cu.ft./min.)	4.927
35	b) Max. Vent End Temp. (deg. F)	655

- c) Time to Max. Temp.
(min.) 9:50
- 7) After Flaming No

Test Results

5 Test results calculated on the basis of the areas under the curves of flame spread distance, temperature, and smoke developed versus time, as shown in Figs. 2a-c, are provided in the table below for calibration materials and for:

10 WOF-24 Pressure Treated
3/4" Particle Board

	<u>Material Description</u>	<u>Flame Spread</u>	<u>Fuel Contributed</u>	<u>Smoke Developed Index</u>
15	High-density inorganic reinforced cement board	0	0	0
	Red oak flooring	100	100	100
20	Test Sample	20	15	0

OBSERVATIONS: No burnthrough. Surface charred and flaky to 9-1/2 feet.

REMARKS: The average moisture content of the first panel was 16.7%.

25 CONCLUSIONS: Based on one test, the flame spread, calculated according to ASTM E-84-94, meets Class A (Class I) - 25 or under flame spread.

Example 3: WOF-24 Pressure-Treated 5/8" Douglas Fir Plywood

30 The Douglas fir plywood test sample was prepared and tested according to the procedures described above. The test results are as follows:

Material Tested

- 1) Manufacturer: World Environmental
Irvington, Alabama
- 35 2) Burn Number 1
- 3) Average Thickness (in.) .616
- 4) Average Weight (lbs./sq.ft.) 2.37

	5)	Average Groove Depth (in.)	N/A
	6)	Product Description: WOF-24 pressure-treated 5/8" Douglas fir plywood Treatment concentration: 33% WOF-24	
5	7)	Color	Brown
	8)	Surface	Both sides treated
	9)	Sample Selection	Manufacturer
	10)	Date of Selection	4/1996
10	11)	Material Description By	Manufacturer
	12)	Method of Mounting	Self-supporting on ledges
	13)	Sample Conditioning (days)	22
		<u>Test Conditions and Data</u>	
15	1)	Specimen Preheat Time (min.)	2:00
	2)	Tunnel Brick Temp. (deg. F)	103
	3)	Ignition Time (seconds)	65
	4)	Time to End of Tunnel or Flamefront Distance	13-1/2' @ 9:30
20	5)	Time-Distance Curve Area (min./ft.)	54.7
	6)	Fuel and Temperature	
	a)	Fuel (cu.ft./min.)	4.933
	b)	Max. Vent End Temp. (deg. F)	670
25	c)	Time to Max. Temp. (min.)	8:05
	7)	After Flaming	No

Test Results

30 Test results calculated on the basis of the areas under the curves of flame spread distance, temperature, and smoke developed versus time, as shown in Figs. 3a-c, are provided in the table below for calibration materials and for:

WOF-24 Pressure Treated
5/8" Douglas Fir Plywood

	<u>Material Description</u>	<u>Flame Spread</u>	<u>Fuel Contributed</u>	<u>Smoke Developed Index</u>
5	High-density inorganic reinforced cement board	0	0	0
10	Red oak flooring	100	100	100
	Test Sample	30	20	15

OBSERVATIONS: No burnthrough. Surface charred and flaky to 12 feet.

REMARKS: The average moisture content of the first panel was 14.1%.

CONCLUSIONS: Based on one test, the flame spread, calculated according to ASTM E-84-94, meets Class B (Class II) - 75 or under flame spread.

The primary object of the present invention is satisfied by the present invention. Prior to the present inventive method, there were no commercially suitable methods for adequately treating green lumber which preserves the lumber and renders the lumber fire, moisture and insect resistant. The present invention is therefore superior for two reasons. First, it provides a method and composition for full penetration of green lumber or other wood products having a high water content as well as providing a superior method and composition for treating dried or manufactured wood products. Second, use of a preservative that penetrates green lumber is commercially advantageous. Prior art pressure treatment methods require that the wood products to be dry before treatment. Therefore, to adequately pressure treat a wood product, the product has to be dried, treated with preservative and re-dried. In contrast, wood products prepared according to the present invention need not be dried prior to treatment,

eliminating the drying step and thereby reducing processing costs, labor costs and energy usage.

The above invention has been described with reference to the preferred embodiment. Obvious
5 modifications and alterations will occur to others upon reading and understanding the preceding detailed description. It is intended that the invention be construed as including all such modifications and alterations insofar as they come within the scope of the
10 appended claims or the equivalents thereof.

I CLAIM:

1. A method for pressure treating wood products comprising the step of pressure treating said wood products with an aqueous solution comprising a silicate and one of a wetting agent and a rheology modifier, wherein the pH of
5 the aqueous solution is at least 9.0.

2. A method for pressure treating wood products as claimed in claim 1, wherein said aqueous solution further comprises borax.

3. A method for pressure treating wood products as claimed in claim 1, wherein said pressure treating step includes the steps of:

- 5 a. placing said wood products in a pressure vessel;
- b. contacting said wood products with said aqueous solution;
- c. increasing said pressure in said pressure vessel;
- 10 d. draining said aqueous solution from said pressure vessel;
- e. reducing said pressure in said pressure vessel; and
- f. drying said wood products.

4. A method for pressure treating wood products as claimed in claim 3, wherein in the step of reducing the pressure in said pressure vessel includes applying a vacuum.

5. A method for pressure treating wood products as claimed in claim 3, further comprising a step of applying a vacuum to the wood products immediately prior to the contacting step.

6. A method for pressure treating wood products as claimed in claim 3, wherein in the step of increasing the pressure, the pressure is increased to 40-160 pounds for 20-90 minutes.

7. A method for pressure treating wood products as claimed in claim 6, wherein the wood products are one of particle board, medium-density fiberboard and oriented strand board and the pressure is increased to about 40
5 pounds.

8. A method for pressure treating wood products as claimed in claim 6, wherein the wood products are one of a natural wood product and green lumber and the pressure is increased to about 140 pounds.

9. A method for pressure treating wood products as claimed in claim 3, wherein in the step of applying the vacuum, a vacuum of approximately 35 pounds is applied.

10. A method for pressure treating wood products as claimed in claim 3, wherein in the step of reducing the pressure in said pressure vessel, the pressure is reduced to about 30 pounds for 10 minutes.

11. A method for pressure treating wood products as claimed in claim 1, wherein the aqueous solution comprises a suitable dye.

12. A method for pressure treating wood products as claimed in claim 1, wherein said aqueous solution comprises 18 to 60% sodium silicate, 0.01 to 2% wetting agent and 0.01 to 2% rheology modifier and water.

13. A method for pressure treating wood products as claimed in claim 12, wherein the solution consists of an aqueous solution of 38% sodium silicate, 0.1% wetting agent and 0.1% rheology modifier, the solution having 21% solids.

14. A method for pressure treating wood products as claimed in claim 12, wherein the solution further comprises 1-10% borax.

15. A solution for pressure treating wood products comprising a silicate and one of a wetting agent and a rheology modifier.

16. A solution for pressure treating wood products as claimed in claim 15, wherein said solution further comprises borax.

17. A solution for pressure treating wood products as claimed in claim 15, wherein the solution includes 18 to 60% sodium silicate, 0.01 to 2% wetting agent and 0.01 to 2% rheology modifier.

18. A solution for pressure treating wood products as claimed in claim 17, wherein said aqueous solution further comprises 1-10% borax.

19. A pressure treated wood product prepared by pressure treating said wood product with an aqueous solution comprising a silicate and one of a wetting agent and a rheology modifier.

20. A pressure treated wood product as claimed in claim 19, including 18-60% sodium silicate and one of 0.1-2% wetting agent, 0.1-2% rheology modifier and 1-10% borax.

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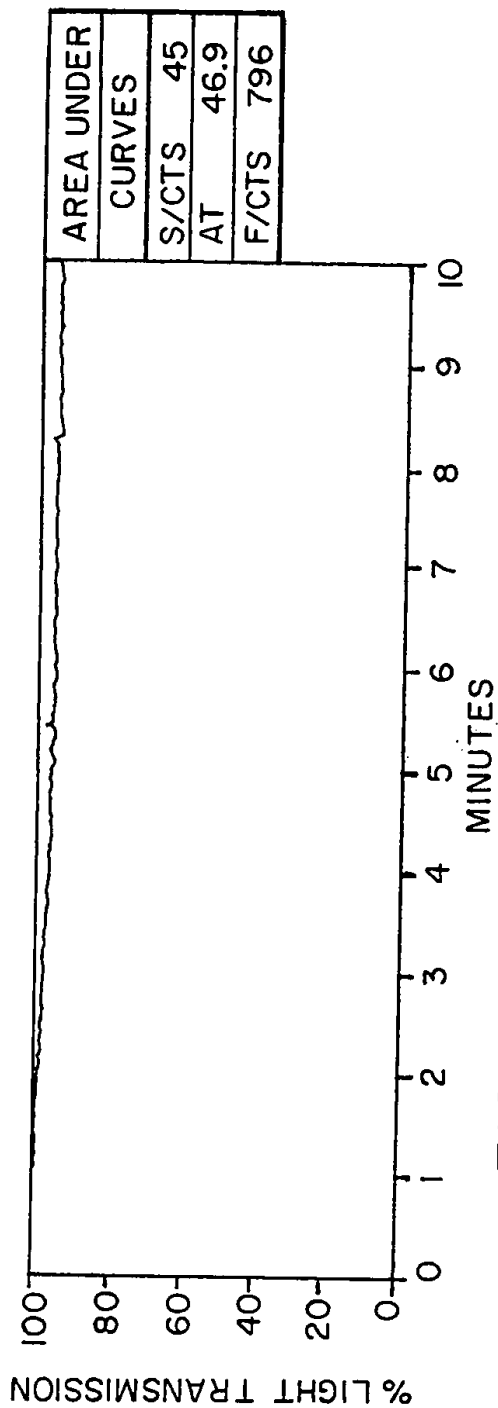


FIG. 1c

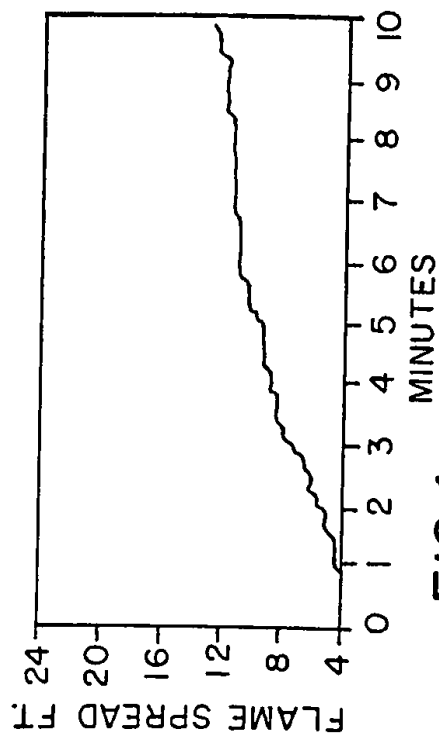


FIG. 1a

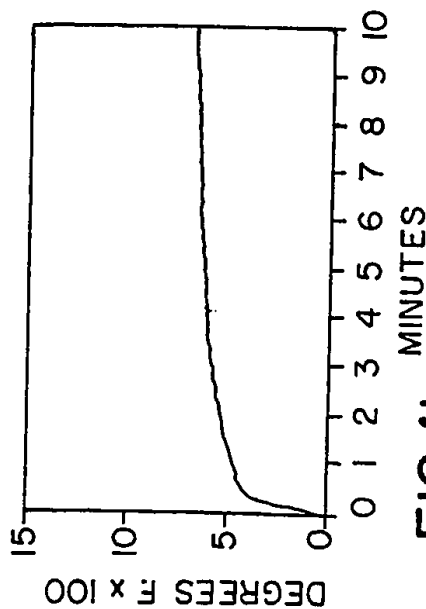


FIG. 1b

2/3

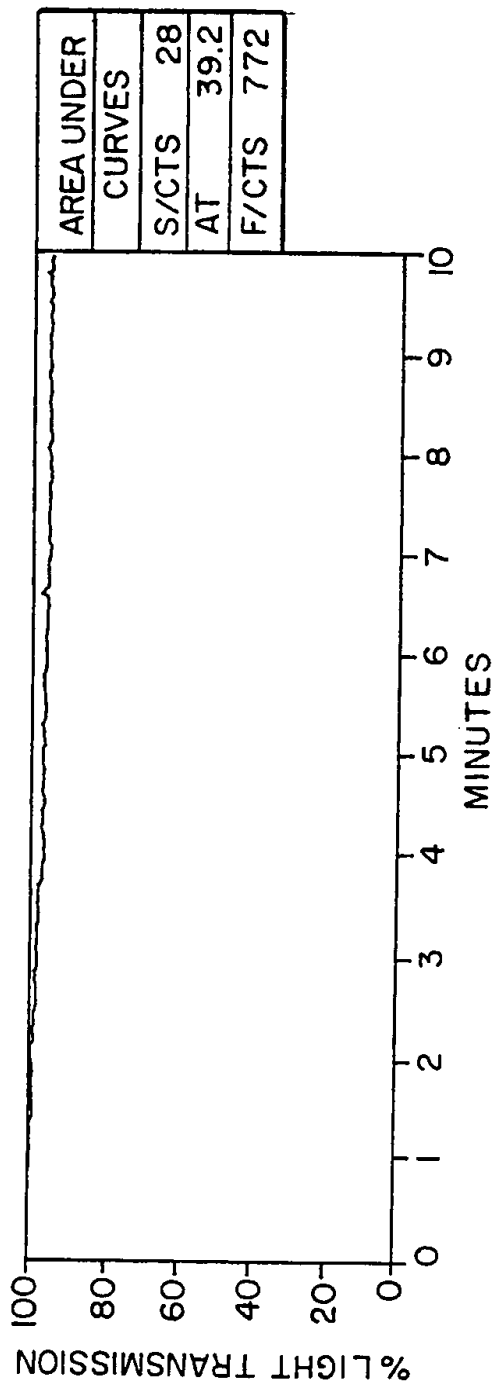


FIG. 2c

SUBSTITUTE SHEET (RULE 26)

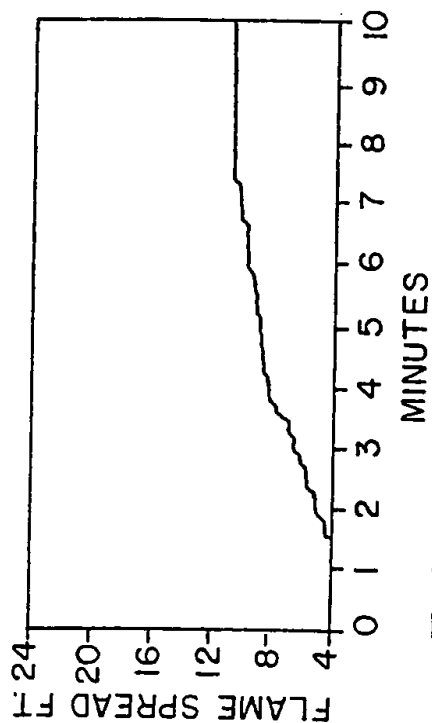


FIG. 2a

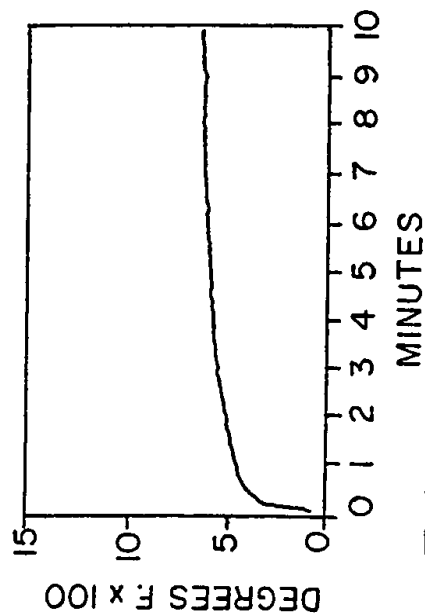


FIG. 2b

3/3

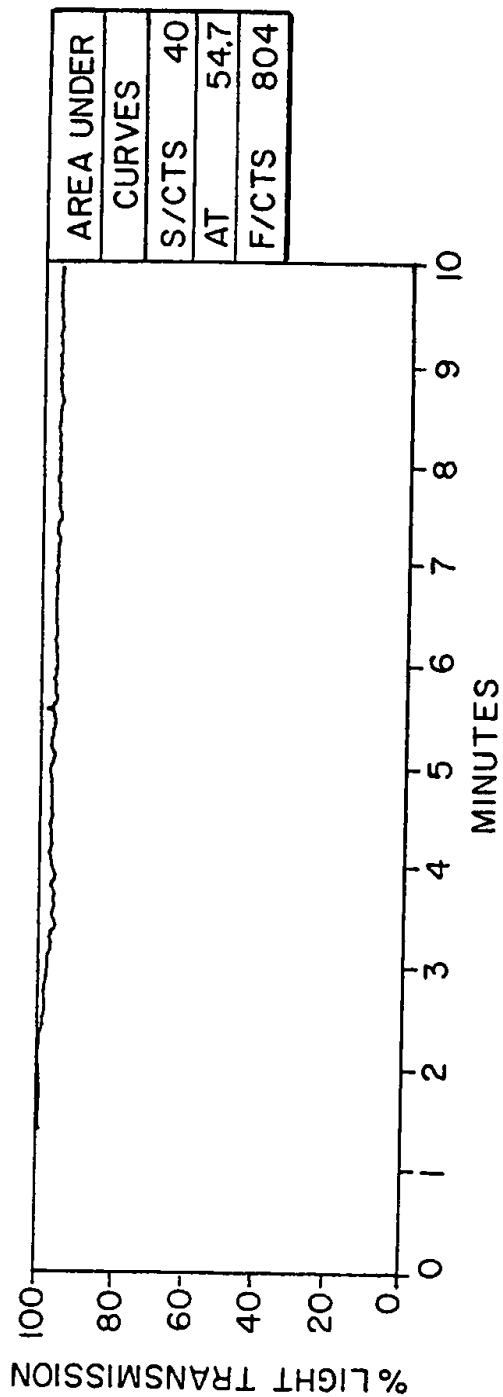


FIG. 3c

SUBSTITUTE SHEET (RULE 26)

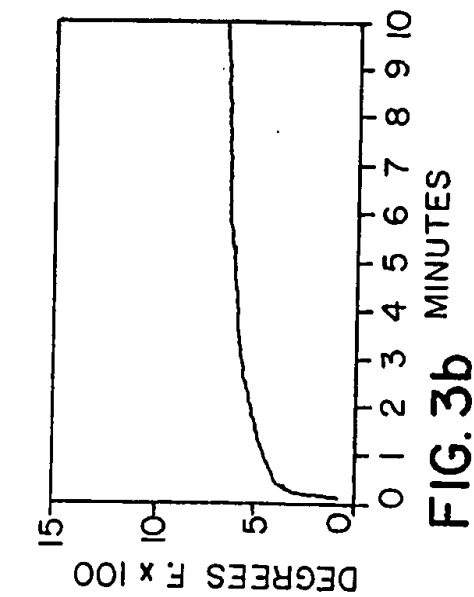


FIG. 3b

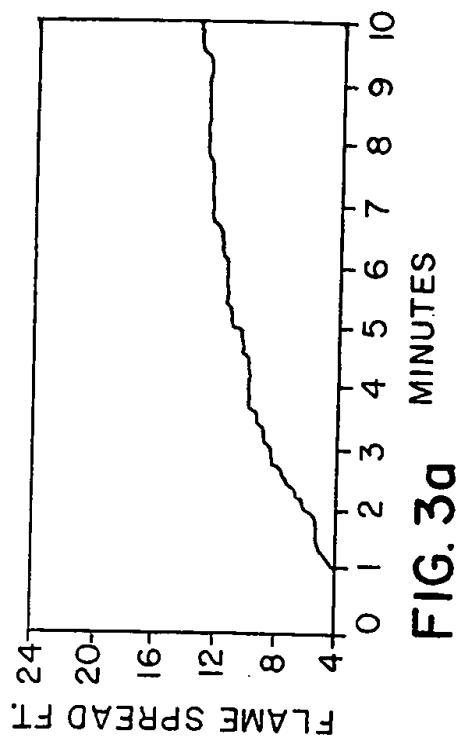


FIG. 3a

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US97/19812

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C09D 15/00; B32B 21/04; B05D 3/00, 3/12, 7/06

US CL : 427/297, 325, 351; 428/537.1; 106/12, 18.12, 18.13

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 427/297, 325, 351; 428/537.1; 106/12, 18.12, 18.13

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NoneElectronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4,542,146 A (VAN GESTEL ET AL) 17 September 1985 (17-09-85), column 1, lines 10-13; column 4, lines 24-60; column 5, lines 10-14.	1-12, 14-20
Y	US 3,968,276 A (ALLEN) 06 July 1976 (06-07-76), column 1, lines 10-20; column 11, line 15 through column 12, line 48.	1-12, 14-20
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☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search
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Date of mailing of the international search report

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Name and mailing address of the ISA/US
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INTERNATIONAL SEARCH REPORT

International application No.
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B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

APS, CAPIus, Japio, WPIDS

search terms: silicate, borax, thickeners, wetting, wood, treatment, impregnate, rheological, pressure, vacuum, sodium borate, sodium silicate, water glass

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

POWER OF ATTORNEY

Docket No.

TPP 30420A

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Title: PROCESS OF USING SODIUM SILICATE TO CREATE FIRE RETARDANT PRODUCTS

Serial No., if Any: 09/927,063

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TO THE ASSISTANT COMMISSIONER FOR PATENTS

The Assistant Commissioner for Patents
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